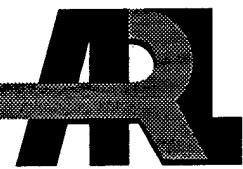


ARMY RESEARCH LABORATORY



# Effects of Coatings on Moisture Absorption in Composite Materials

by James F. Newill, Steven H. McKnight,  
Christopher P. R. Hoppel, and Gene R. Cooper

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ARL-TR-2099

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# **Army Research Laboratory**

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**James F. Newill, Steven H. McKnight, Christopher P. R. Hoppel,  
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Weapons and Materials Research Directorate, ARL

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## **Abstract**

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Polymeric coatings can be used to slow the ingress of moisture in composite materials. The effectiveness of these coatings is limited by the quality and thickness, as well as the moisture diffusivity and solubility. The present study investigates the long-term effectiveness of coatings for thick composite laminates, as well as traditional thin laminates. The results are compared for static and fluctuating boundary conditions, with comparisons showing how theoretical predictions can be used to define the coating performance characteristics for protecting composite structures.

The theoretical approach is used to determine important attributes for moisture protection of polymer-matrix composite structures provided by polymeric coatings. Developed solutions are used to evaluate a range of coating materials and thicknesses for thin and thick substrates. The diffusion constants and the saturation levels for the coating material were obtained from the range of available coating materials. The results include typical diffusion patterns for coating materials, saturation of the coating layer, development of moisture through time, and effects of the material properties for the various substrate thickness. Moisture diffusion behavior at the substrate-coating interface is also presented. For relatively thick coating layers, the results show that the diffusion constant and the saturation level of the coating must both be low to significantly affect the diffusion process.

## **Acknowledgements**

The authors would like to thank Dr. William Drysdale (U.S. Army Research Laboratory) for his technical guidance throughout this project. This study was supported by Office of Program Manager, Tank and Medium Caliber Main Armament Systems (OPM-TMAS). Funding for Dr. Steve McKnight was provided in part by SERDP under Pollution Prevention Program #1133 "Mechanisms of Military Coatings Degradation." This funding is greatly appreciated.

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## Table of Contents

	<u>Page</u>
<b>ACKNOWLEDGEMENTS.....</b>	iii
<b>TABLE OF CONTENTS .....</b>	v
<b>LIST OF FIGURES .....</b>	vii
<b>LIST OF TABLES.....</b>	ix
<b>1. INTRODUCTION.....</b>	1
<b>2. METHODOLOGY.....</b>	2
<b>3. TYPICAL RESULTS.....</b>	7
3.1. CONSTANT BOUNDARY CONDITION.....	7
3.2. TRANSIENT BOUNDARY CONDITIONS .....	10
<b>4. CONCLUSIONS .....</b>	18
<b>5. REFERENCES.....</b>	19
<b>DISTRIBUTION LIST .....</b>	21
<b>REPORT DOCUMENTATION PAGE .....</b>	33

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## List of Figures

<u>Figure</u>	<u>Page</u>
1. Schematic of Problem Description .....	2
2. Experimental Setup for FTIR-ATR Diffusion Measurement.....	5
3. Results From a Typical FTIR-ATR for Polyurethane.....	6
4. Diffusion Results for $k = 2$ , $a = 1$ , $l_s = 0.5$ , and $l_c = 0.01$ in .....	8
5. Effect of Coatings on the Diffusion Potential at the Mid-Plane ( $l_s = 0.1$ in, $l_c = 0.01$ in).....	8
6. Diffusion Potential at the Mid-Plane for $l_s = 0.1$ in and $l_c = 0.002$ in and $l_c = 0.01$ in, Respectively. ....	9
7. Relative Humidity for the Full Cycle and for 24 hr.....	10
8. The Moisture Distribution of the Coated and Uncoated Substrate After 90 Days and at the Mid-Plane vs. Time.....	10
9. $l_s = 0.1$ in, $l_c = 0.01$ in, $a = 0.5$ , $k = 10$ .....	11
10. $l_s = 0.1$ in, $l_c = 0.01$ in, $a = 0.5$ , $k = 0.1$ .....	12
11. $l_s = 0.1$ in, $l_c = 0.01$ in, $a = 2$ , $k = 0.1$ .....	12
12. $l_s = 0.1$ in, $l_c = 0.05$ in, $a = 0.5$ , $k = 10$ .....	13
13. $l_s = 0.1$ in, $l_c = 0.05$ in, $a = 0.5$ , $k = 0.1$ .....	13
14. $l_s = 0.1$ in, $l_c = 0.05$ in, $a = 2$ , $k = 0.1$ .....	14
15. $l_s = 0.25$ in, $l_c = 0.01$ in, $a = 0.5$ , $k = 10$ .....	14
16. $l_s = 0.25$ in, $l_c = 0.01$ in, $a = 0.5$ , $k = 0.1$ .....	15
17. $l_s = 0.25$ in, $l_c = 0.05$ in, $a = 0.5$ , $k = 0.1$ .....	15
18. $l_s = 0.25$ in, $l_c = 0.05$ in, $a = 2$ , $k = 0.1$ .....	16
19. Urethane Paint on Epoxy Primer. ....	16
20. Mid-Plane Response of Urethane Paint.....	17

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## List of Tables

<u>Table</u>	<u>Page</u>
1. Diffusion Coefficients and Equilibrium Weight Gain for Coatings. ....	6

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## 1. Introduction

Moisture absorption is a significant design consideration for polymer-matrix composites. For U.S. Army applications, the hygroscopic expansion of composite materials and the subsequent effects on dimensional stability are extremely important (Newill et al. 1999). Typical ordnance structures are made of thick (on the order of several hundred plies), precise, high-quality composite parts. These structures are designed with very tight dimensional tolerances; 120-mm tank ammunition is designed to have between 0.1-mm and 0.2-mm clearance between the outer diameter of the bullet and the inner diameter of the gun barrel. Small changes in the diameters have been shown to affect the in-bore performance of the rounds. These structures must be capable of long-term storage and deployment in a variety of environmental conditions. Therefore, understanding the absorption of moisture and the expansion of the structures in realistic environments is essential for determining the maximum exposure times and conditions for the ammunition.

The present study uses solutions developed for analyzing moisture diffusion in multidomain structures subject to transient boundary conditions. While diffusion in polymers and polymeric coatings can be defect driven, this paper takes a theoretical approach to characterize the effectiveness of ideal (no cracks or pinholes) coatings on composites. This allows the basic material properties of the coating and substrates, as well as the coating thickness, to be evaluated analytically to focus experimental work on the actual materials and processes. The theoretical work can then be used to extend predictions to complicated structures and realistic long-term environments (Springer 1977; Newill, Hoppel, and Berman 1998).

It is convenient to begin moisture diffusion analysis by assuming Fickian diffusion for the moisture uptake for a single homogenous material. Also, if the sample thickness is much less than the length and width, the diffusion can be considered one-dimensional. Polymers eventually reach maximum moisture content for a given surrounding humidity level. The relation between the maximum moisture content and the external humidity level is given by,

$$M_m = \alpha(R.H.)^\beta, \quad (1)$$

where  $M_m$  is the maximum moisture content,  $\alpha$  and  $\beta$  are material constants, and  $R.H.$  is the surrounding relative humidity level (Tsai 1988; Pipes, Vinson, and Chou 1976; Shen and Springer 1976).

## 2. Methodology

This study investigates theoretical diffusion performance of coated substrates. It is intended to help focus experimental work and provide estimates of the “best case” moisture protection that can be afforded by coatings. Figure 1 shows the schematic of the problem. The problem is divided into two regions, the substrate, and the coating. The substrate is the material that is being protected from moisture intrusion. The purpose of this paper is to investigate some of the geometric parameters and material properties that govern moisture diffusion.

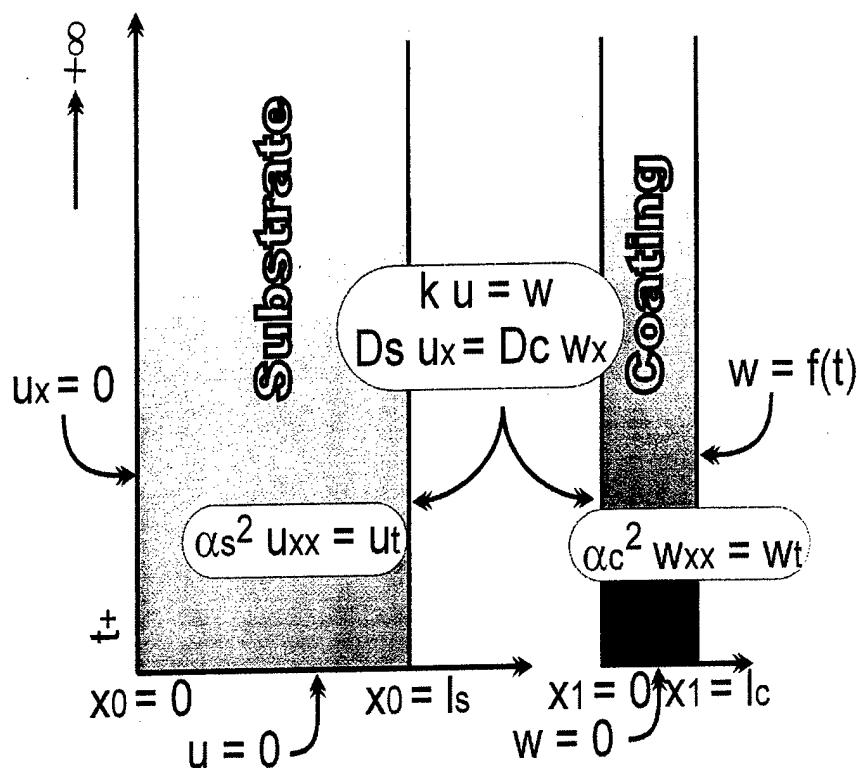


Figure 1. Schematic of Problem Description.

The solution for the moisture content of the substrate ( $u$ ) and the coating ( $w$ ) is given in equations (2) and (3) (Newill et al., to be published) with the applicable constants defined in equations (4) through (10) and the roots of the equations defined in equation (11):

$$u(x,t) = -4 \int_0^t \frac{f(t-\tau)}{(k+1/\sqrt{a})\Psi_1} \sum_{n=1}^{\infty} \frac{\sqrt{s_n} \cos(\sqrt{s_n} x_0/l_s) e^{-s_n t}}{\sin(\sqrt{s_n} \Psi_1(1+\varepsilon))(1+\varepsilon) + \phi \sin(\sqrt{s_n} \Psi_1(1-\varepsilon))(1-\varepsilon)} d\tau, \quad (2)$$

$$w(x,t) = -4 \int_0^t \left[ \frac{\frac{f(t-\tau)\sqrt{s_n}}{\Psi_1(k+1/\sqrt{a})}}{\sum_{n=1}^{\infty} \frac{\frac{1}{\sqrt{a}} \sin(\sqrt{s_n} \Psi_1) \sin\left(\frac{\sqrt{s_n} x_1/l_s}{\sqrt{a}}\right) - k \cos(\sqrt{s_n} \Psi_1) \cos\left(\frac{\sqrt{s_n} x_1/l_s}{\sqrt{a}}\right)}{\sin(\sqrt{s_n} \Psi_1(1+\varepsilon))(1+\varepsilon) + \phi \sin(\sqrt{s_n} \Psi_1(1-\varepsilon))(1-\varepsilon)}} e^{-s_n t} \right] d\tau, \quad (3)$$

$$\phi = \frac{k-1/\sqrt{a}}{k+1/\sqrt{a}}, \quad (4)$$

$$\Psi_1 = \frac{l_s}{\alpha_s}, \quad (5)$$

$$\varepsilon = \frac{1}{c\sqrt{a}}, \quad (6)$$

$$a = \frac{D_c}{D_s} = \frac{\alpha_c^2}{\alpha_s^2}, \quad (7)$$

$$\sqrt{a} = \frac{\alpha_c}{\alpha_s}, \quad (8)$$

$$k = \frac{Sat_c}{Sat_s}, \quad (9)$$

$$c = \frac{l_s}{l_c}, \quad (10)$$

$$s_n \text{'s are the roots of } \cos(\sqrt{s_n} \Psi_1(1+\varepsilon)) + \phi \cos(\sqrt{s_n} \Psi_1(1-\varepsilon)) = 0. \quad (11)$$

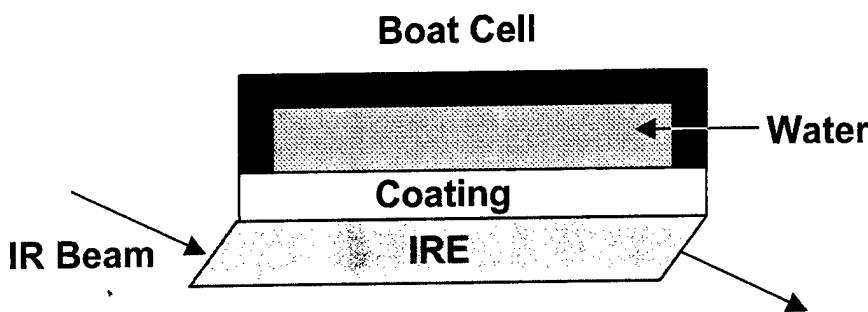
Figure 1 also defines the interface and boundary conditions used for solution. The solution is divided into two domains, each with its own local coordinate system. It is assumed that both domains start without any moisture. At the right side of the domain, equation (1) is used for  $f(t)$  where " $\alpha$ " is assumed to be the saturation level of the coating material and " $\beta$ " is assumed to be 1 (Tsai 1988). At the interface, two conditions must be met. First, the driving potential, % moisture/saturation, of the two materials must be the same; second, the flux at the interface must be continuous (Carslaw 1959).

There are two important ratios present in this problem. They are the ratio of the saturation levels, " $k$ " and the ratio of the diffusion rates of the coating material to the substrate, " $a$ " equation (4). The range of values used for " $a$ " and " $k$ " in this study were obtained from reviewing the moisture literature and data from the coating manufacturer. The range of diffusion rates for coating materials was from an order of magnitude below the diffusion rate for the substrates (T650/1914:  $D = 7.479E-06 \text{ in}^2/\text{hr}$  at  $101^\circ \text{ F}$ ; AS4/Ultem:  $D = 4.396E-05 \text{ in}^2/\text{hr}$  at  $101^\circ \text{ F}$ ) (Bogetti et al. 1997) to an order of magnitude above. The range for the saturation level of the coating materials was from 0.25 times to 8 times the saturation levels for the substrates (e.g., T650/1914's saturation level is 0.76%; AS4/Ultem's saturation level is 0.44%). This does not imply that there is a coating that represents every combination of " $a$ " and " $k$ " used in the study.

In order to provide meaningful predictions, accurate material data must exist for the coatings and the composites of interest. These data include diffusion coefficients, solubility, and directional dependence of moisture transport (if any). Traditional methods for determining the rate of diffusion of permeants in polymers generally employ gravimetric means. The techniques are very difficult to use on thin samples such as polymeric coatings. Other experimental techniques can be used to study diffusion in thin polymer coatings. Fourier transform infrared-attenuated total reflectance (FTIR-ATR) spectroscopy is a very effective method for penetrant diffusivity evaluation (Fieldson and Barbari 1993; Nguyen, Byrd, and Lin 1991; McKnight and Gillespie 1997).

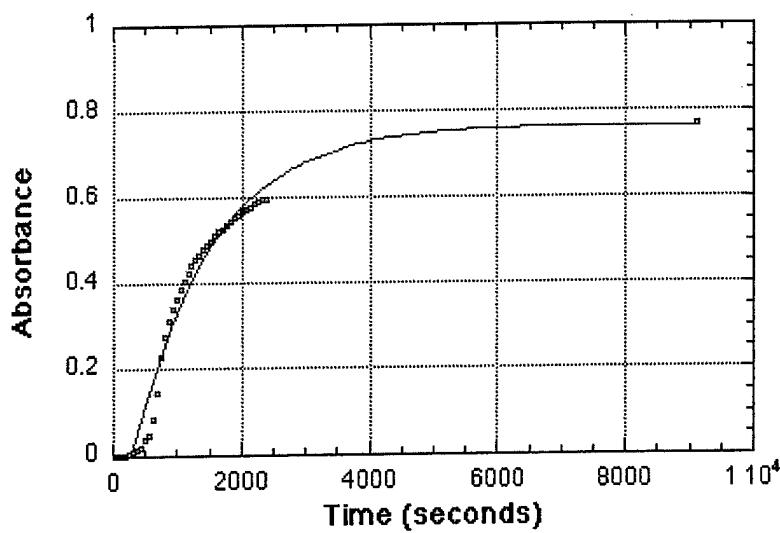
FTIR-ATR studies on diffusion of water through organic coatings have been established, and the experimental details have been reported in the literature (Fieldson and Barbari 1993; Nguyen, Byrd, and Lin 1991; McKnight and Gillespie 1997). Briefly, a coating is applied to an

internal reflection element (IRE) and a “boat cell” is placed adjacent to the coated IRE. The penetrant is injected into the boat cell and diffuses through the coating, and the IR spectra are monitored in real time. Figure 2 shows the setup schematically. The measured absorbance can be directly related to the concentration of the absorbing species, and the diffusion coefficient can be determined using a suitable diffusion model (Fieldson and Barbari 1993; McKnight and Gillespie 1997).



**Figure 2. Experimental Setup for FTIR-ATR Diffusion Measurement.**

In this work, germanium IREs ( $n = 4.0$ ) were coated with military-type coatings. A polyurethane and epoxy coating was evaluated. Experiments were performed using liquid water as the permeant to reflect immersion conditions. A Fickian data reduction scheme was used to calculate the diffusion coefficients. To determine the equilibrium moisture content, free-standing coating films were saturated with water and then dried in a thermogravimetric analyzer (TGA), which measured weight loss with time. Results from a typical FTIR-ATR experiment are shown in Figure 3. Table 1 summarizes the diffusion constants and equilibrium weight gain.



**Figure 3. Results From a Typical FTIR-ATR for Polyurethane.**

**Table 1. Diffusion Coefficients and Equilibrium Weight Gain for Coatings.**

Polymer	Temp. (°C)	Diff. Coef. (cm <sup>2</sup> /s)	Final Uptake (weight-percent)	Reference
Epoxy/Amine	25	5.3E-8	4.25	This Work
Polyurethane CARC	25	1.2E-8	8.20	This Work
Polyetherimide	25	3.0E-9	--	Bogetti (1997)
Polyacrylonitrile	25	1.0E-10	--	Fieldson (1993)

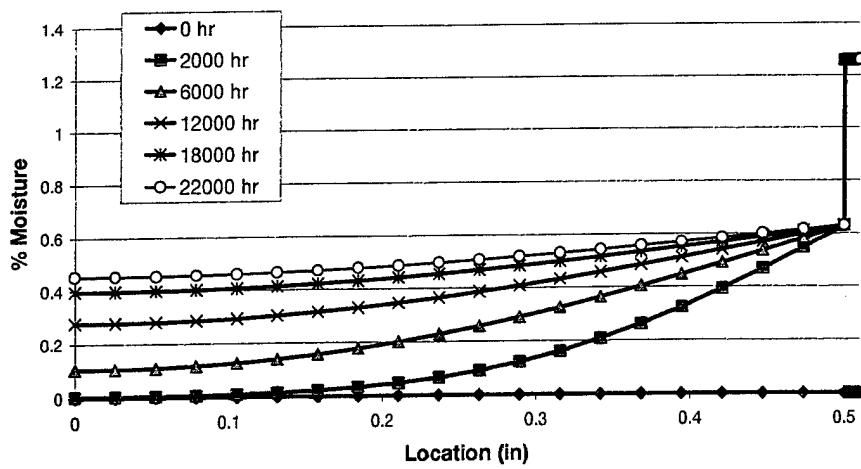
### **3. Typical Results**

Results are presented for two types of cases. The first case uses constant boundary conditions, and the second case uses transient boundary conditions. The constant boundary conditions results are presented to give basic insight into the process; and, the transient results are used to assess long-term protection to a realistic environment. The solutions are used coupled with material data to predict the ability of coatings to protect substrates.

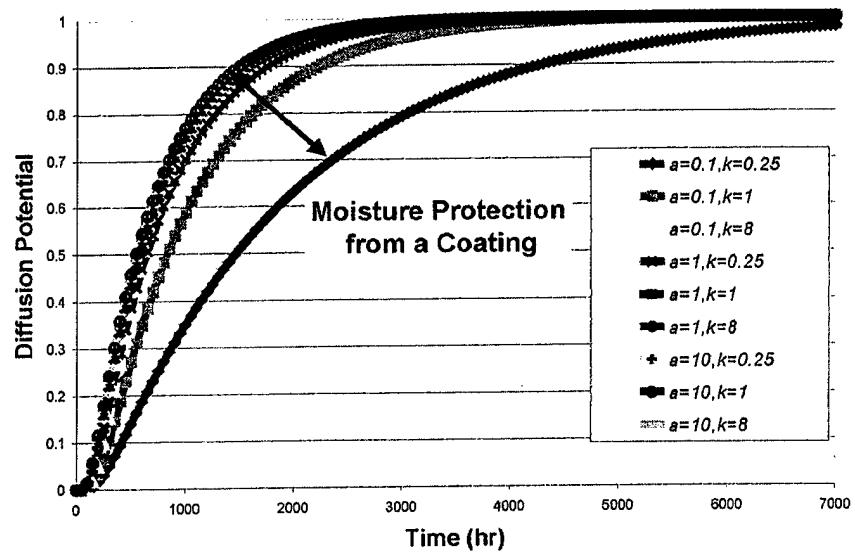
#### **3.1. Constant Boundary Condition**

Figure 4 shows typical results for coated substrates subjected to constant boundary conditions. There are several important features. First, at the interface between the coating and substrate, there is a discontinuity. The discontinuity is a function of the difference in the saturation levels of the coating and substrate. Although it is difficult to see in the figure, the flux at the interface is also matched.

The normalized moisture level at the mid-plane of the substrate was used as a measure of moisture penetration. The moisture level was normalized by dividing by the saturation level of substrate and is referred to as the diffusion potential. As a comparison, the equivalent substrate without a coating is shown to gauge relative diffusion performance. The substrate with  $\alpha = 1$  and  $k = 1$  implies that the coating layer has the same diffusion rate and saturation level as the substrate. For each of the analyses, material properties were chosen for the substrate, then the coating material properties were varied relative to the substrates. Figure 5 shows the general effects of coatings.



**Figure 4.** Diffusion Results for  $k = 2$ ,  $a = 1$ ,  $l_s = 0.5$ , and  $l_c = 0.01$  in.

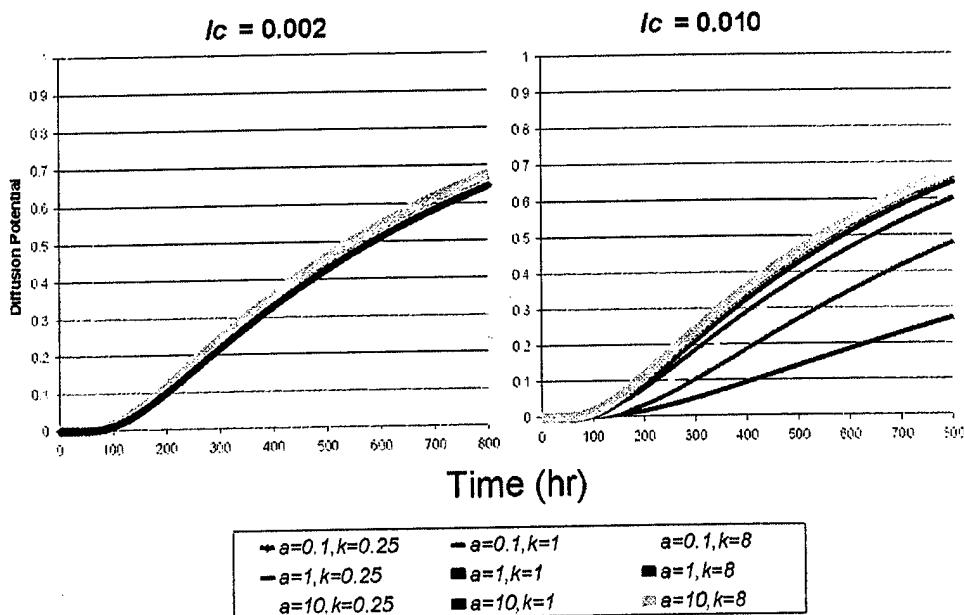


**Figure 5.** Effect of Coatings on the Diffusion Potential at the Mid-Plane ( $l_s = 0.1$  in,  $l_c = 0.01$  in).

Since each line in the figure represents a different type of coating, the separations between the lines imply the particular coatings effect. Each of the lines should be gauged against the case where  $a$  and  $k$  are equal to 1, which represents the effect of just thickening the substrate without adding a coating.

The effectiveness of a coating with constant boundary conditions should be made at a time corresponding to the separation near the largest deviation seen in the Figure 5, which is the best-

case performance for the coating. Figure 6 shows the diffusion potential (which is percent moisture/saturation value which ranges from 0 to 1) results for coating thickness from  $lc = 0.002$  in. to  $lc = 0.01$  in.



**Figure 6. Diffusion Potential at the Mid-Plane for  $ls = 0.1$  in and  $lc = 0.002$  in and  $lc = 0.01$  in, Respectively.**

In Figure 6, some very small differences can be seen around 800 hr. The maximum value remains near 0.66, and it is important to note that the coated substrates do not appreciably perform differently than the uncoated substrates. In Figure 6, with  $lc = 0.01$  in, a dramatic difference can be seen in the diffusion performance of the different coatings. Even with this relatively thick coating, five of the nine combinations of "a" and "k" do not cause the coated substrate to behave differently from the uncoated substrate. This shows that, if the material properties of the coating are chosen properly and the coating is applied thick enough, the diffusion can be cut in half. It appears from these figures that control of both "a" and "k" are necessary to affect diffusion. This is not surprising as the total permeability of the coating can be expected to be a product of the diffusivity and solution of moisture in the coating.

### 3.2. Transient Boundary Conditions

Next, the moisture absorption of the substrate is investigated when protected by a coating and subjected to transient boundary conditions. The relative humidity system use is described in Figure 7.

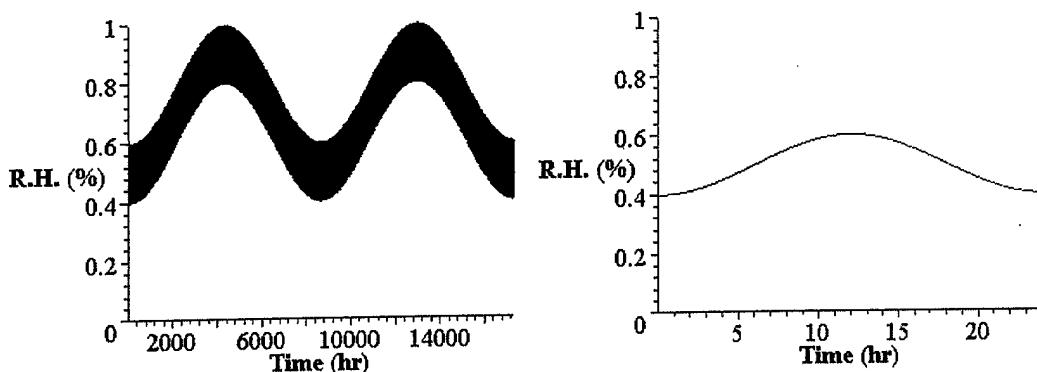


Figure 7. Relative Humidity for the Full Cycle and for 24 hr.

This cycle is similar to data taken in the field in Army vehicles. The cycle is extrapolated over two years. Figure 7 also shows a typical cycle for a 24-hr period.

Figure 8 shows the response of the coated substrate to the transient cycle described in Figure 7.

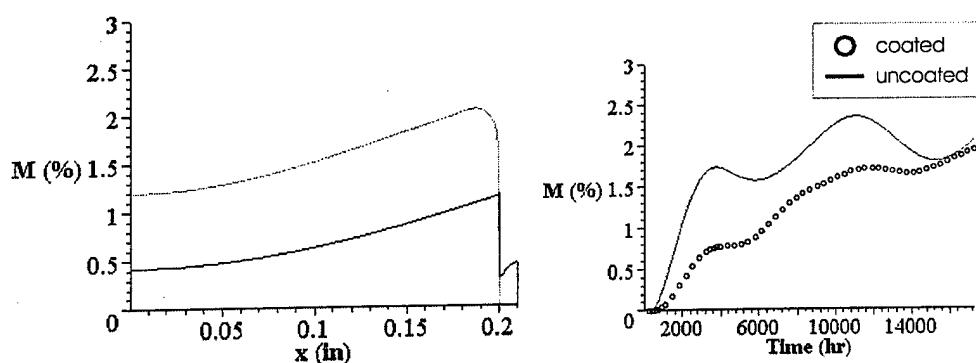


Figure 8. The Moisture Distribution of the Coated and Uncoated Substrate After 90 Days and at the Mid-Plane vs. Time.

The substrate material used for these figures is typical high performance Gr/Ep ( $3.3 \times 10^{-6}$  in $^2$ /hr, saturation 0.4%). Figure 8 shows some differences when compared to constant

boundary condition case (Figure 4). The differences seen between these figures are at the coating interface and at the boundary for the uncoated case. Figure 8 also shows the moisture content at the mid-plane of the substrate.

From Figures 9 through 18, a range of different moisture responses can be seen for the different coatings. Figure 9 shows that the coating had no effect, and Figures 11 and 12 show only a small difference between the performance of the coating and the uncoated substrate as in many other cases. Only in three of the cases did the coating provide some level of protection to the substrate (Figures 10, 13, and 14). It should be noted that, in two of these cases, the coating provided only short- and medium-term protection. In the long term, the moisture distributions of the coated and uncoated layer were the same. For these three cases, the saturation level of the coating was only 10% of the saturation of the substrate. The diffusion rate had less effect. Figures 10 and 14 indicate that changing the diffusion rate by a factor of 4 gives similar results. Figure 13 shows that the combination of low relative diffusion rate coupled with low relative saturation level and a thick coating yields the best protection. Figures 15 through 18 show the same scenarios for a substrate with a thickness of 0.25 in. These results are almost identical, again showing that it is the combination of the coating relatively low saturation level, relative low diffusion rate, and sufficient thickness are needed to protect a substrate. The combination of the relative low diffusion rate and thicker coating also act to change the phase of the response.

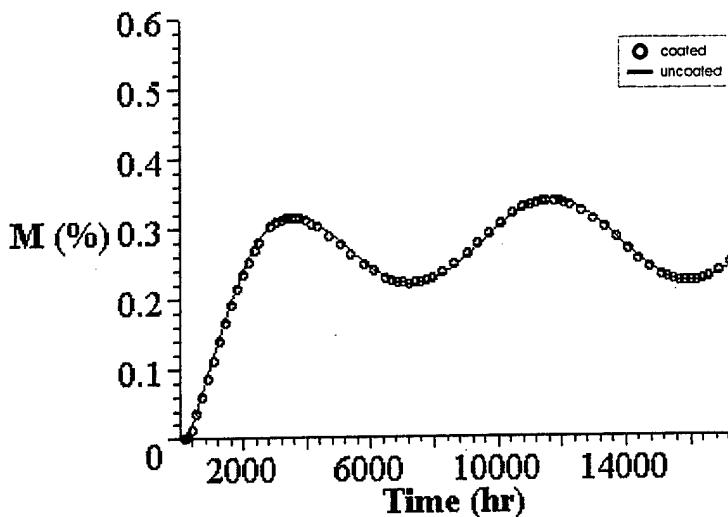


Figure 9.  $ls = 0.1$  in,  $lc = 0.01$  in,  $a = 0.5$ ,  $k = 10$ .

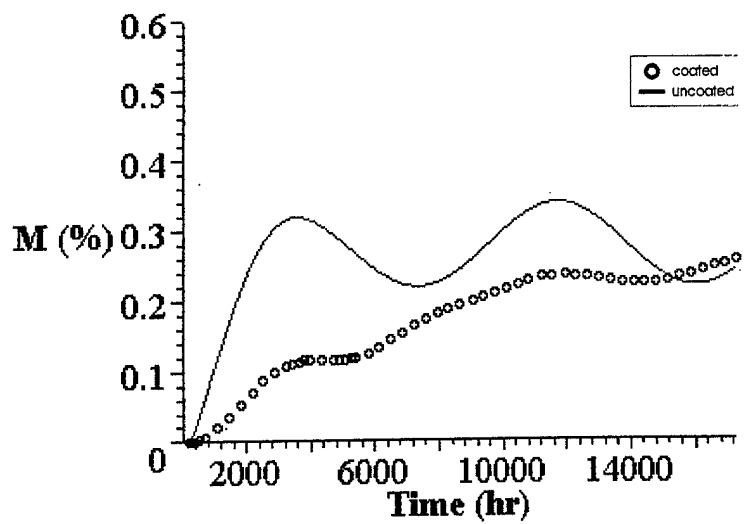


Figure 10.  $ls = 0.1$  in,  $lc = 0.01$  in,  $a = 0.5$ ,  $k = 0.1$ .

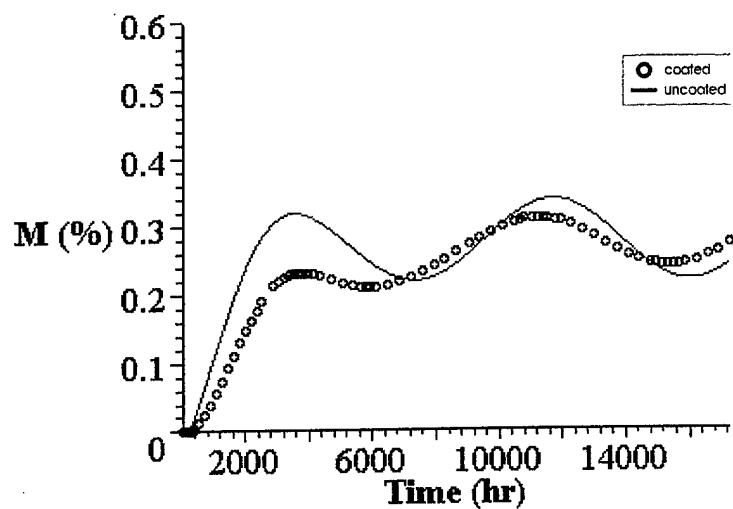
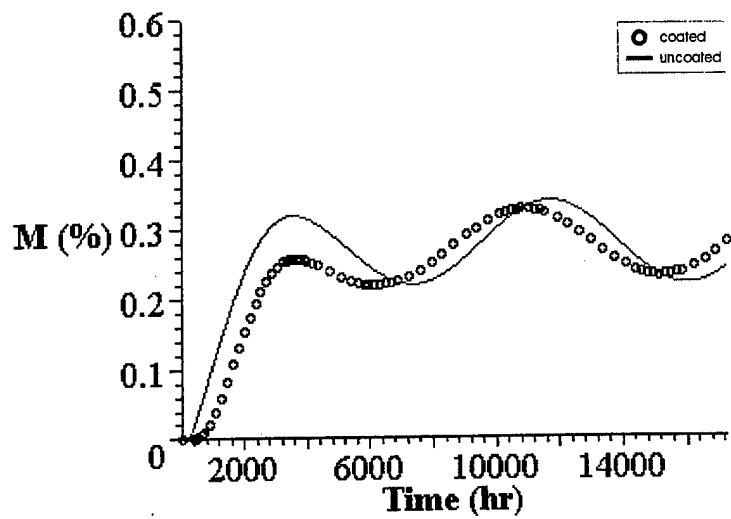
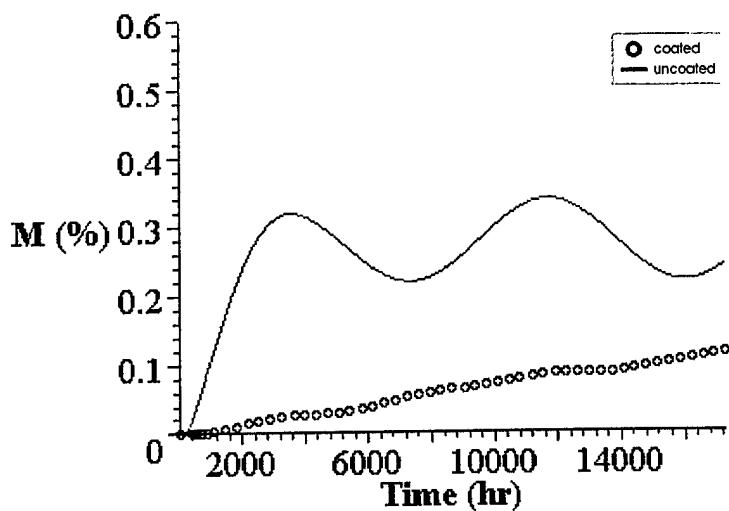


Figure 11.  $ls = 0.1$  in,  $lc = 0.01$  in,  $a = 2$ ,  $k = 0.1$ .



**Figure 12.**  $ls = 0.1$  in,  $lc = 0.05$  in,  $a = 0.5$ ,  $k = 10$ .



**Figure 13.**  $ls = 0.1$  in,  $lc = 0.05$  in,  $a = 0.5$ ,  $k = 0.1$ .

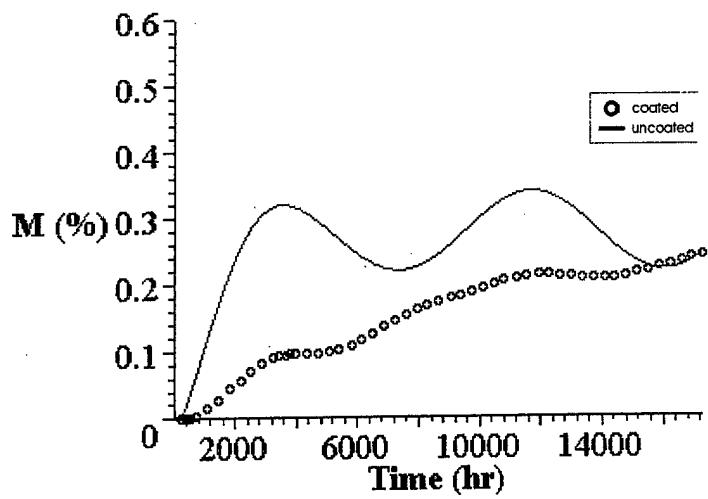


Figure 14.  $ls = 0.1$  in,  $lc = 0.05$  in,  $a = 2$ ,  $k = 0.1$ .

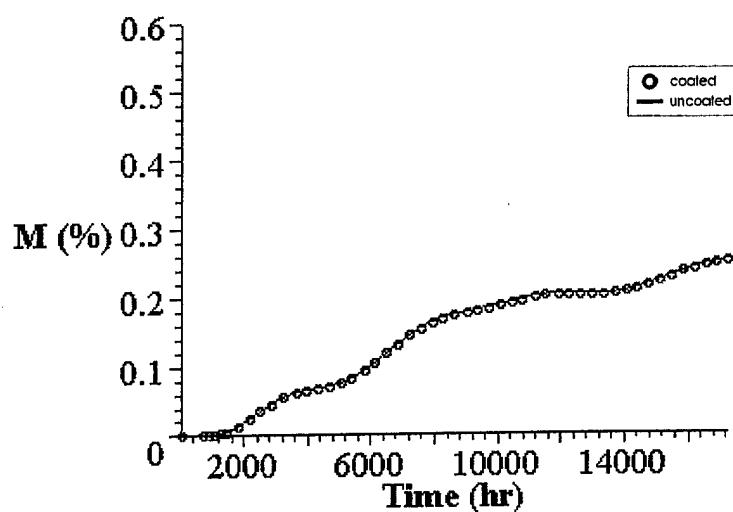


Figure 15.  $ls = 0.25$  in,  $lc = 0.01$  in,  $a = 0.5$ ,  $k = 10$ .

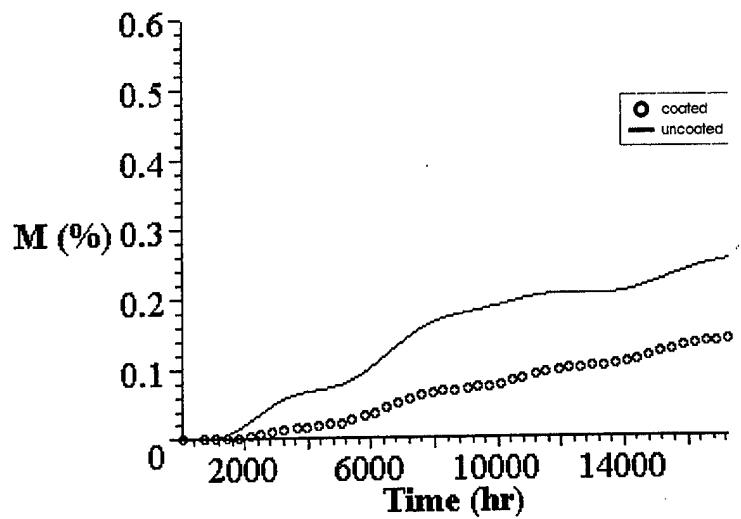


Figure 16.  $ls = 0.25$  in,  $lc = 0.01$  in,  $a = 0.5$ ,  $k = 0.1$ .

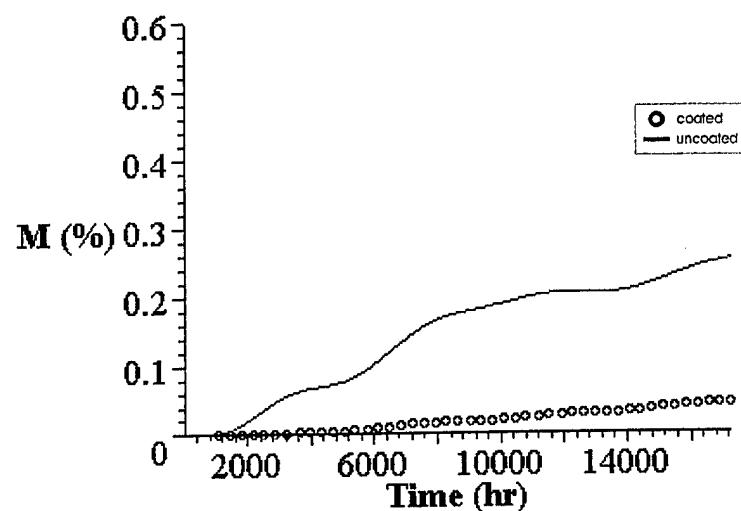


Figure 17.  $ls = 0.25$  in,  $lc = 0.05$  in,  $a = 0.5$ ,  $k = 0.1$ .

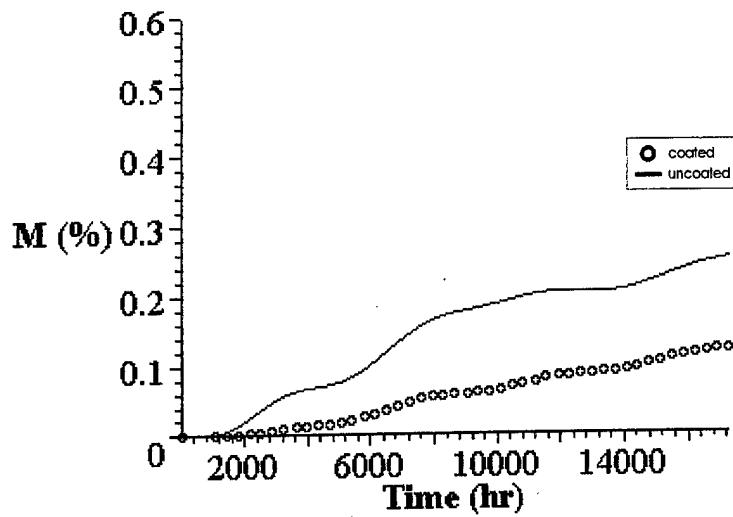


Figure 18.  $s = 0.25$  in,  $lc = 0.05$  in,  $a = 2$ ,  $k = 0.1$ .

A final example considers 0.002 in epoxy primer substrate that is coated with 0.005 in urethane paint. Figure 19 shows the paint saturates in hours yielding very little moisture protection while Figure 20 show difference between uncoated epoxy primer and the epoxy primer with the urethane paint. The difference between the moisture content in the primer with and without the paint is nearly identical.

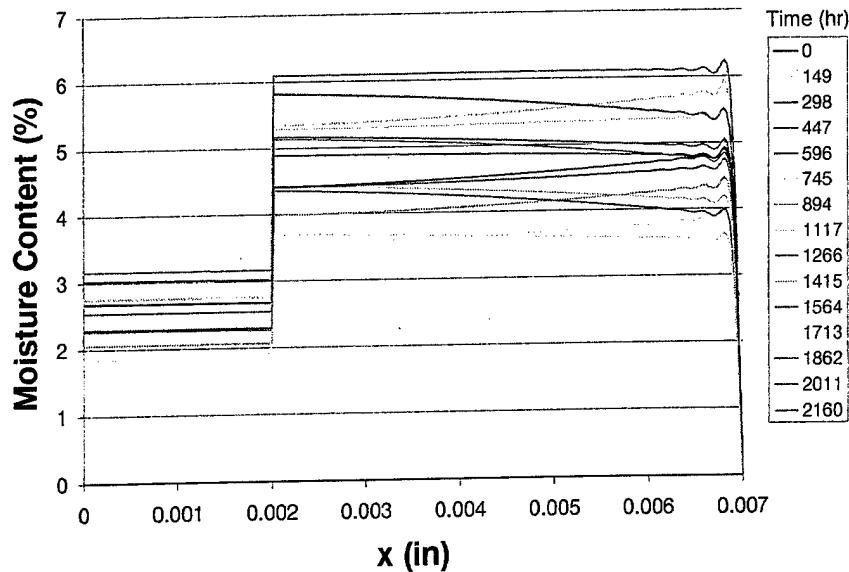
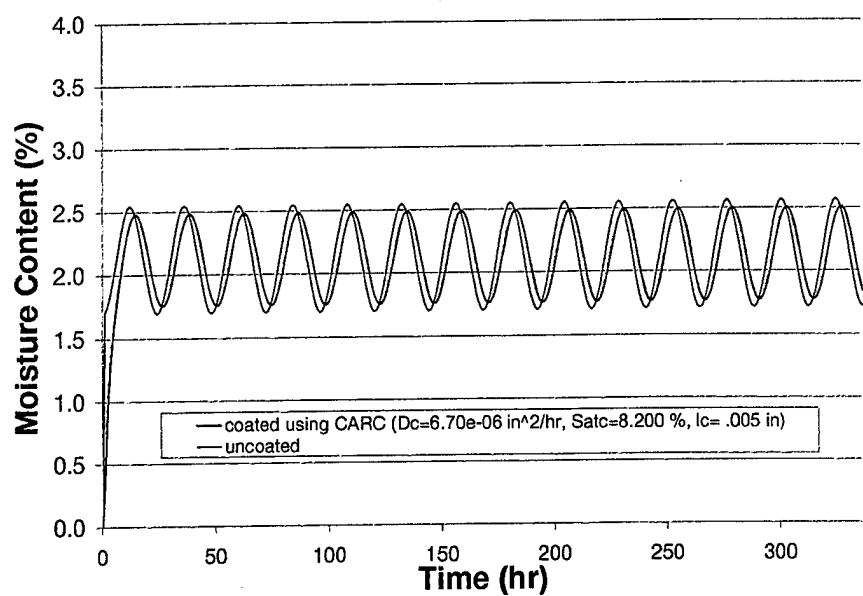


Figure 19. Urethane Paint on Epoxy Primer.



**Figure 20. Mid-Plane Response of Urethane Paint.**

## **4. Conclusions**

Polymeric coatings can be used to reduce the moisture absorption in polymer and polymer-composite structures. The analytical approach taken in this paper demonstrates the thickness, diffusion constant, and saturation level of the coating relative to those of the substrate are essential to the coatings effectiveness. The diffusion rate and the saturation level of the coating must both be very small compared to the substrate for the coating to affect the long-term diffusion behavior. Even when the material constants are optimized, the coating must be relatively thick to affect a substantial reduction of moisture absorption. Even with coatings thickness 10% of the substrate thickness or larger, many values of "a" and "k" are not effective in protecting the structure from moisture ingress.

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<b>13. ABSTRACT (Maximum 200 words)</b>  <p>Polymeric coatings can be used to slow the ingress of moisture in composite materials. The effectiveness of these coatings is limited by the quality and thickness, as well as the moisture diffusivity and solubility. The present study investigates the long-term effectiveness of coatings for thick composite laminates, as well as traditional thin laminates. The results are compared for static and fluctuating boundary conditions, with comparisons showing how theoretical predictions can be used to define the coating performance characteristics for protecting composite structures.</p> <p>The theoretical approach is used to determine important attributes for moisture protection of polymer-matrix composite structures provided by polymeric coatings. Developed solutions are used to evaluate a range of coating materials and thicknesses for thin and thick substrates. The diffusion constants and the saturation levels for the coating material were obtained from the range of available coating materials. The results include typical diffusion patterns for coating materials, saturation of the coating layer, development of moisture through time, and effects of the material properties for the various substrate thickness. Moisture diffusion behavior at the substrate-coating interface is also presented. For relatively thick coating layers, the results show that the diffusion constant and the saturation level of the coating must both be low to significantly affect the diffusion process.</p>			
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